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(*Z*)-3-Benzyl-1,5-benzothiazepin-4(5*H*)-one

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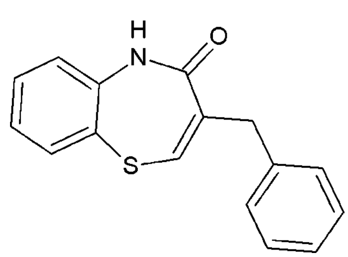
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.036; wR factor = 0.100; data-to-parameter ratio = 14.4.

In the crystal structure of the title compound, $C_{16}H_{13}NOS$, molecules are linked into cyclic centrosymmetric $R_2^2(8)$ dimers *via* pairs of N—H···O hydrogen bonds. The seven-membered ring adopts a boat conformation.

Related literature

For the pharmaceutical properties of thiazepin derivatives, see: Tomascovic *et al.* (2000); Rajsner *et al.* (1971); Metys *et al.* (1965). For conformations of thiazepin derivatives, see: Huang *et al.* (2011). For a related structure, see: Sabari *et al.* (2012).For graph-set analysis of hydrogen bonds, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_{16}H_{13}NOS$
M_r = 267.33
Monoclinic, $P2_1/c$
a = 9.3409 (6) Å
b = 11.7876 (7) Å
c = 11.8310 (6) Å
β = 94.727 (3)°
V = 1298.24 (13) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 293 K
0.32 × 0.20 × 0.10 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
T_{min} = 0.980, T_{max} = 0.990
11696 measured reflections
3220 independent reflections
2700 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.100
S = 1.03
3220 reflections
224 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H···O1^{i}	0.847 (18)	2.098 (19)	2.9407 (15)	173.1 (16)

Symmetry code: (i) $-x+1, -y+1, -z+2$.

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5942).

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supplementary materials

Acta Cryst. (2012). E**68**, o2329 [doi:10.1107/S1600536812029054]

(*Z*)-3-Benzyl-1,5-benzothiazepin-4(5*H*)-one

V. Sabari, R. Selvakumar, M. Bakthadoss and S. Aravindhan

Comment

The title compound is used as an intermediate for the synthesis of dosulepin, which is an antidepressant of the tricyclic family. Dosulepin prevents reabsorbing of serotonin and noradrenaline in the brain, helps to prolong the mood lightening effect of any released noradrenaline and serotonin, thus relieving depression. The dibenzo[c,e]thiazepin derivatives exhibit chiroptical properties (Tomascovic *et al.*, 2000). Dibenzo[b,e]thiazepin-5,5-dioxide derivatives possess antihistaminic and antiallergenic activities (Rajsner *et al.*, 1971). Benzene thiazepin derivatives are identified as a new type of effective antihistaminic compounds (Metys *et al.*, 1965). Considering the wide range of biological activities of the thiazepin derivatives, we determined the crystal structure of the title compound.

X-Ray analysis confirms the molecular structure and atom connectivity as illustrated in Fig. 1. The seven membered thiazepin ring adopts a boat conformation (Huang *et al.*, 2011). The atom O1 deviates by 1.0431 (11) Å from the least-squares plane of the thiazepin ring. The sum of the bond angles around the N1 atom (357.73°) of the thiazepin ring is in agreement with sp^2 hybridization. The molecules are linked into cyclic centrosymmetric dimers *via* N—H···O hydrogen bonds with the motif $R_2^2(8)$ (Bernstein *et al.*, 1995).

Experimental

A mixture of (*Z*)-methyl 2-(bromomethyl)-3-phenylacrylate (2 mmol) and *o*-aminothiophenol (2 mmol) in the presence of potassium *tert*-butoxide (4.8 mmol) in dry THF (10 ml) was stirred at room temperature for 1 h. After the completion of the reaction as indicated by TLC, the reaction mixture was concentrated and the resulting crude mass was diluted with water (20 ml) and extracted with ethyl acetate (3 *x* 20 ml). The organic layer was washed with brine (2 *x* 20 ml) and dried over anhydrous sodium sulfate. The organic layer was concentrated, which successfully provide the crude final product ((*Z*)-3-benzylbenzo[*b*][1,4]thiazepin-4(5*H*)-one). The final product was purified by column chromatography on silica gel to afford the title compound 41% yields.

Refinement

H atoms were freely refined.

Computing details

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT* (Bruker, 2008); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008) and *PLATON* (Spek, 2009).

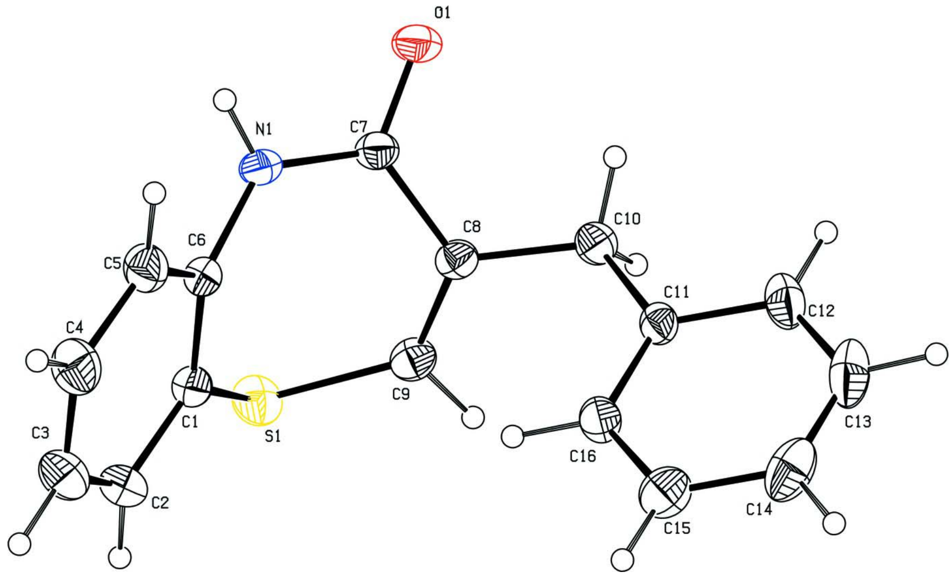

Figure 1

The molecular structure of the title compound, showing 30% probability displacement ellipsoids.

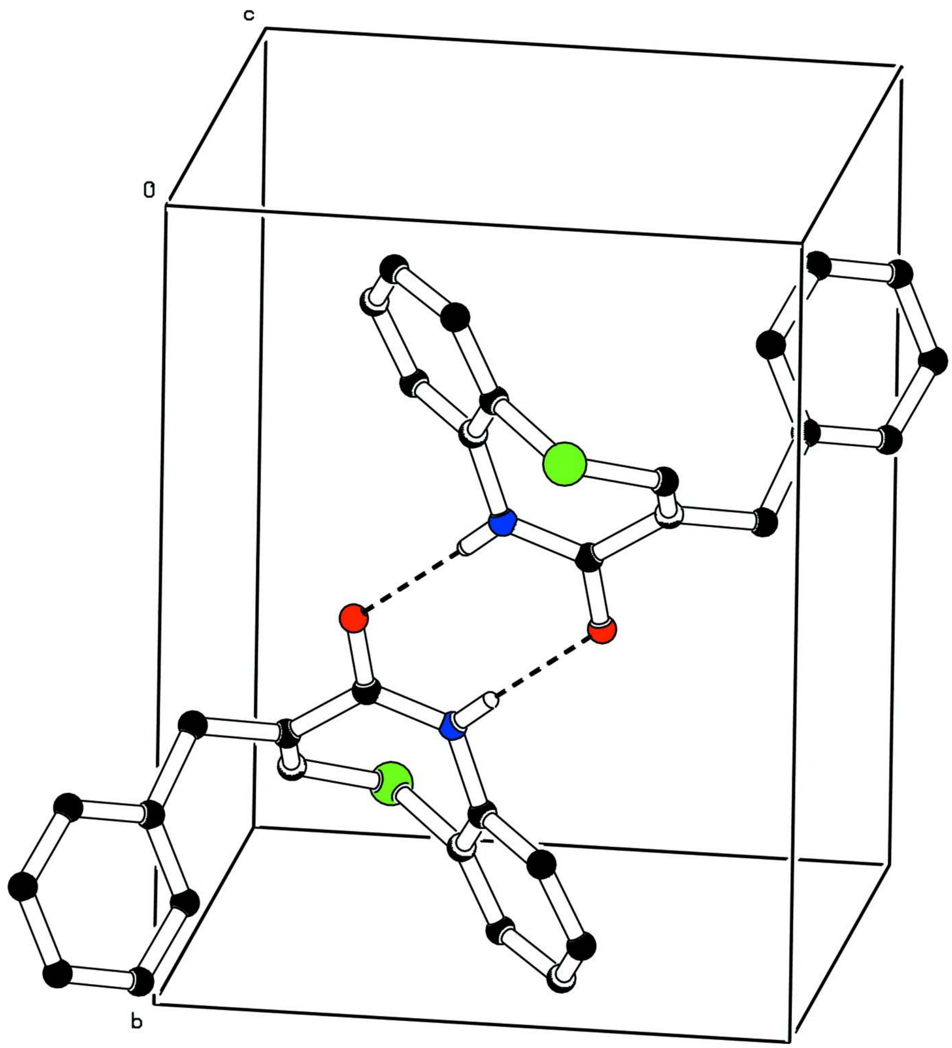

Figure 2

A view of the crystal packing. H atoms not involved in hydrogen bonding (dashed lines) have been omitted for clarity.

(*Z*)-3-Benzyl-1,5-benzothiazepin-4(5*H*)-one

Crystal data

$C_{16}H_{13}NOS$
M_r = 267.33
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 9.3409 (6) Å
b = 11.7876 (7) Å
c = 11.8310 (6) Å
β = 94.727 (3)°
V = 1298.24 (13) Å^3
Z = 4
$F(000)$ = 560
D_x = 1.368 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 8725 reflections
θ = 2.8–29.1°
μ = 0.24 mm^{-1}
T = 293 K
Monoclinic, colourless
0.32 × 0.20 × 0.10 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 15.9948 pixels mm^{-1}
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
$T_{min} = 0.980$, $T_{max} = 0.990$
11696 measured reflections
3220 independent reflections
2700 reflections with $I > 2\sigma(I)$
$R_{int} = 0.024$
$\theta_{max} = 28.3°$, $\theta_{min} = 2.2°$
$h = -12 \rightarrow 12$
$k = -12 \rightarrow 15$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.100$
$S = 1.03$
3220 reflections
224 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0473P)^2 + 0.3853P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.31$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
H10A	0.9319 (19)	0.4199 (16)	1.0576 (15)	0.054 (5)*
H14	1.158 (2)	0.0216 (19)	1.2706 (18)	0.071 (6)*
H13	1.253 (3)	0.210 (2)	1.287 (2)	0.090 (7)*
H2	0.520 (2)	0.0349 (17)	0.7510 (18)	0.067 (6)*
H4	0.264 (2)	0.0652 (19)	1.0082 (18)	0.075 (6)*
H3	0.341 (2)	−0.0401 (19)	0.8584 (18)	0.078 (6)*
H5	0.367 (2)	0.2445 (15)	1.0477 (16)	0.051 (5)*
H9	0.906 (2)	0.2452 (13)	0.8225 (15)	0.047 (4)*
H12	1.152 (2)	0.3537 (18)	1.1720 (16)	0.066 (6)*
H16	0.8605 (18)	0.1324 (14)	1.0263 (14)	0.048 (4)*
H15	0.960 (2)	−0.0149 (19)	1.1441 (18)	0.075 (6)*
H	0.4802 (19)	0.4003 (15)	0.9656 (14)	0.050 (5)*
H10B	1.0195 (19)	0.3709 (15)	0.9641 (15)	0.053 (5)*
S1	0.67169 (4)	0.23482 (4)	0.74334 (3)	0.04629 (13)
O1	0.69525 (11)	0.49768 (9)	1.00015 (11)	0.0515 (3)
N1	0.54541 (12)	0.35755 (9)	0.94400 (10)	0.0376 (3)

C9	0.81284 (15)	0.27217 (12)	0.84256 (12)	0.0377 (3)
C1	0.54547 (14)	0.18101 (11)	0.83286 (11)	0.0361 (3)
C8	0.80771 (13)	0.33382 (10)	0.93653 (11)	0.0326 (3)
C7	0.67881 (14)	0.40033 (11)	0.96316 (11)	0.0345 (3)
C10	0.94322 (15)	0.35387 (12)	1.01201 (13)	0.0397 (3)
C6	0.49997 (14)	0.24447 (10)	0.92206 (11)	0.0332 (3)
C11	0.99742 (13)	0.25642 (11)	1.08739 (11)	0.0335 (3)
C16	0.94145 (15)	0.14763 (12)	1.08123 (12)	0.0396 (3)
C12	1.11413 (16)	0.27689 (16)	1.16571 (13)	0.0490 (4)
C5	0.39623 (16)	0.20069 (14)	0.98728 (14)	0.0448 (3)
C14	1.11727 (19)	0.08314 (19)	1.22626 (15)	0.0599 (5)
C13	1.17296 (18)	0.1913 (2)	1.23380 (15)	0.0628 (5)
C2	0.48567 (18)	0.07488 (14)	0.80956 (15)	0.0511 (4)
C15	1.00050 (19)	0.06142 (15)	1.15013 (14)	0.0508 (4)
C4	0.33594 (18)	0.09600 (16)	0.96179 (17)	0.0568 (4)
C3	0.37969 (19)	0.03362 (15)	0.87283 (18)	0.0613 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0532 (2)	0.0562 (2)	0.02983 (18)	−0.00076 (17)	0.00540 (15)	−0.00396 (14)
O1	0.0427 (6)	0.0312 (5)	0.0809 (8)	0.0002 (4)	0.0071 (5)	−0.0154 (5)
N1	0.0322 (6)	0.0297 (5)	0.0512 (7)	0.0043 (4)	0.0054 (5)	−0.0075 (5)
C9	0.0372 (7)	0.0390 (7)	0.0381 (7)	0.0021 (5)	0.0096 (5)	0.0012 (5)
C1	0.0350 (6)	0.0356 (7)	0.0370 (6)	0.0033 (5)	−0.0019 (5)	−0.0029 (5)
C8	0.0327 (6)	0.0275 (6)	0.0382 (6)	−0.0004 (5)	0.0067 (5)	0.0038 (5)
C7	0.0363 (6)	0.0280 (6)	0.0396 (6)	0.0018 (5)	0.0054 (5)	−0.0013 (5)
C10	0.0339 (7)	0.0322 (7)	0.0530 (8)	−0.0054 (5)	0.0037 (6)	−0.0020 (6)
C6	0.0301 (6)	0.0311 (6)	0.0377 (6)	0.0021 (5)	−0.0018 (5)	−0.0018 (5)
C11	0.0258 (6)	0.0411 (7)	0.0340 (6)	−0.0021 (5)	0.0057 (5)	−0.0062 (5)
C16	0.0374 (7)	0.0402 (7)	0.0403 (7)	−0.0027 (6)	−0.0011 (5)	0.0001 (6)
C12	0.0338 (7)	0.0670 (10)	0.0457 (8)	−0.0105 (7)	0.0010 (6)	−0.0089 (7)
C5	0.0367 (7)	0.0492 (8)	0.0489 (8)	−0.0044 (6)	0.0063 (6)	−0.0045 (7)
C14	0.0478 (9)	0.0839 (13)	0.0477 (9)	0.0169 (9)	0.0033 (7)	0.0187 (9)
C13	0.0374 (8)	0.1029 (16)	0.0461 (9)	−0.0014 (9)	−0.0081 (7)	0.0013 (9)
C2	0.0507 (9)	0.0421 (8)	0.0593 (9)	0.0009 (7)	−0.0025 (7)	−0.0175 (7)
C15	0.0534 (9)	0.0482 (9)	0.0509 (8)	0.0051 (7)	0.0045 (7)	0.0096 (7)
C4	0.0458 (9)	0.0543 (10)	0.0705 (11)	−0.0141 (7)	0.0066 (8)	0.0015 (8)
C3	0.0529 (10)	0.0412 (9)	0.0888 (13)	−0.0142 (7)	−0.0001 (9)	−0.0104 (9)

Geometric parameters (Å, º)

S1—C9	1.7479 (15)	C11—C12	1.392 (2)
S1—C1	1.7663 (14)	C16—C15	1.388 (2)
O1—C7	1.2332 (16)	C16—H16	0.972 (17)
N1—C7	1.3460 (17)	C12—C13	1.377 (3)
N1—C6	1.4165 (16)	C12—H12	0.97 (2)
N1—H	0.846 (18)	C5—C4	1.379 (2)
C9—C8	1.3324 (19)	C5—H5	0.942 (19)
C9—H9	0.976 (18)	C14—C13	1.377 (3)

C1—C2	1.388 (2)	C14—C15	1.380 (3)
C1—C6	1.3885 (18)	C14—H14	0.96 (2)
C8—C7	1.4920 (17)	C13—H13	0.96 (3)
C8—C10	1.5064 (19)	C2—C3	1.378 (3)
C10—C11	1.515 (2)	C2—H2	0.92 (2)
C10—H10A	0.958 (19)	C15—H15	0.98 (2)
C10—H10B	0.968 (18)	C4—C3	1.373 (3)
C6—C5	1.387 (2)	C4—H4	0.97 (2)
C11—C16	1.3845 (19)	C3—H3	0.95 (2)

C9—S1—C1	101.05 (6)	C11—C16—C15	121.13 (14)
C7—N1—C6	129.99 (11)	C11—C16—H16	118.4 (10)
C7—N1—H	114.0 (12)	C15—C16—H16	120.4 (10)
C6—N1—H	113.8 (12)	C13—C12—C11	121.01 (17)
C8—C9—S1	128.37 (11)	C13—C12—H12	120.7 (12)
C8—C9—H9	118.4 (10)	C11—C12—H12	118.2 (12)
S1—C9—H9	113.2 (10)	C4—C5—C6	120.17 (15)
C2—C1—C6	119.54 (13)	C4—C5—H5	121.1 (11)
C2—C1—S1	118.81 (11)	C6—C5—H5	118.7 (11)
C6—C1—S1	121.57 (10)	C13—C14—C15	119.21 (16)
C9—C8—C7	123.22 (12)	C13—C14—H14	122.2 (13)
C9—C8—C10	119.61 (12)	C15—C14—H14	118.5 (13)
C7—C8—C10	116.46 (12)	C14—C13—C12	120.67 (16)
O1—C7—N1	119.66 (12)	C14—C13—H13	121.2 (15)
O1—C7—C8	119.00 (12)	C12—C13—H13	118.1 (15)
N1—C7—C8	121.30 (11)	C3—C2—C1	120.36 (15)
C8—C10—C11	116.97 (11)	C3—C2—H2	122.7 (13)
C8—C10—H10A	109.6 (11)	C1—C2—H2	117.0 (13)
C11—C10—H10A	109.4 (11)	C14—C15—C16	120.12 (17)
C8—C10—H10B	107.9 (10)	C14—C15—H15	120.0 (12)
C11—C10—H10B	106.0 (11)	C16—C15—H15	119.9 (13)
H10A—C10—H10B	106.3 (15)	C3—C4—C5	120.30 (16)
C5—C6—C1	119.61 (13)	C3—C4—H4	119.7 (13)
C5—C6—N1	117.44 (12)	C5—C4—H4	120.0 (13)
C1—C6—N1	122.71 (12)	C4—C3—C2	119.98 (16)
C16—C11—C12	117.84 (14)	C4—C3—H3	119.7 (13)
C16—C11—C10	124.38 (12)	C2—C3—H3	120.2 (13)
C12—C11—C10	117.75 (13)		

C1—S1—C9—C8	−49.95 (14)	C7—N1—C6—C1	−56.6 (2)
C9—S1—C1—C2	−128.63 (12)	C8—C10—C11—C16	8.3 (2)
C9—S1—C1—C6	54.52 (12)	C8—C10—C11—C12	−173.77 (12)
S1—C9—C8—C7	−11.3 (2)	C12—C11—C16—C15	−0.6 (2)
S1—C9—C8—C10	178.70 (10)	C10—C11—C16—C15	177.38 (14)
C6—N1—C7—O1	−167.26 (14)	C16—C11—C12—C13	0.8 (2)
C6—N1—C7—C8	15.0 (2)	C10—C11—C12—C13	−177.31 (14)
C9—C8—C7—O1	−135.97 (15)	C1—C6—C5—C4	−1.7 (2)
C10—C8—C7—O1	34.35 (18)	N1—C6—C5—C4	172.74 (14)
C9—C8—C7—N1	41.8 (2)	C15—C14—C13—C12	−0.6 (3)

C10—C8—C7—N1	−147.91 (13)	C11—C12—C13—C14	−0.2 (3)
C9—C8—C10—C11	−76.59 (17)	C6—C1—C2—C3	1.4 (2)
C7—C8—C10—C11	112.72 (14)	S1—C1—C2—C3	−175.52 (14)
C2—C1—C6—C5	0.5 (2)	C13—C14—C15—C16	0.8 (3)
S1—C1—C6—C5	177.32 (11)	C11—C16—C15—C14	−0.2 (2)
C2—C1—C6—N1	−173.63 (13)	C6—C5—C4—C3	1.0 (3)
S1—C1—C6—N1	3.20 (18)	C5—C4—C3—C2	0.9 (3)
C7—N1—C6—C5	129.13 (16)	C1—C2—C3—C4	−2.1 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H···O1^{i}	0.847 (18)	2.098 (19)	2.9407 (15)	173.1 (16)

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+2.